

**A CRITICAL EVALUATION OF THE FACTORS INFLUENCING
HIGH STRAIN RATE SUPERPLASTICITY**

FINAL PROGRESS REPORT

TERENCE G. LANGDON

JANUARY 31, 2000

U.S. ARMY RESEARCH OFFICE

GRANT NUMBER DAAH04-96-1-0332

UNIVERSITY OF SOUTHERN CALIFORNIA

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Summary of the results obtained on this program

This research program was initiated in August 1996 with the two objectives of (1) conducting detailed experiments to provide a detailed understanding of the creep behavior of the metal matrix composites which are characteristic of high strain rate superplasticity (HSR SP) and (2) to endeavor to use a new processing technique, known as Equal-Channel Angular Pressing (ECAP), to achieve submicrometer and nanometer grain sizes in structural alloys and to use these materials to provide the first demonstration of HSR SP in conventional materials at relatively low temperatures. Both of these objectives were achieved during the life-time of this program and in the summer of 1999 the program was continued on a no-cost extension for an additional 6 months, up to January 2000, in order to provide an opportunity to bring the research to a satisfactory conclusion.

During the 3.5 years of this research program, a total of 47 papers was either submitted to the scientific literature or submitted for publication in conference proceedings. A complete listing of all of these papers is given in this report. Reprints of all publications have been submitted to ARO as they became available during the course of the program except only for publications #46 and #47 on the attached list which are currently in press and have been submitted only as preprints. There has been much interest in this work within the scientific community, especially in our use of the ECAP procedure, and in this respect it should be noted that the following 10 publications on the attached list are the publications arising from invited talks at national or international conferences: #7, 10, 14, 18, 21, 25, 31, 32, 41 and 47.

Full details of the results are given in the many publications documented herein. Briefly, the major findings in the first objective of this program may be summarized as follows:

- (a) Metal matrix composites exhibit a creep behavior incorporating a threshold stress which can be easily deduced using a new extrapolation procedure.
- (b) The creep rates within the metal matrix composites are governed by creep processes occurring within the matrix alloy.
- (c) The creep rates in the composite may be influenced by the occurrence of a load transfer which serves to transfer some of the applied load to the reinforcement.
- (d) By incorporating both load transfer and substructure strengthening into the analysis, it is possible to provide a consistent interpretation of both high temperature creep and high strain rate superplasticity and, in particular, it is possible to explain the anomalously high activation energies which are a consistent feature of HSR SP.
- (e) Consistent results can be attained between powder metallurgy and ingot metallurgy metals when the threshold stresses are correctly incorporated into the analyses.
- (f) The so-called substructure-invariant model for creep of metal matrix composites does not provide consistent results and must be discarded in favor of an interpretation of creep in terms of conventional climb and glide processes.

The second objective of this program led to remarkable success and, even within this short time-frame, a very clear demonstration that ECAP is a viable processing tool for achieving exceptionally small grain sizes and unusual material properties. The major findings of this part of the program may be summarized as follows:

- (a) Equal-channel angular pressing is capable of achieving very substantial grain refinement, typically to the submicrometer level, in a wide range of materials.
- (b) The ultrafine grains introduced by ECAP are formed in the first pass through the ECAP die in the form of subgrains separated by low angle boundaries, but subsequently these subgrain boundaries evolve into high angle boundaries as more dislocations enter the subgrain walls.
- (c) In simple materials, such as pure metals and solid solution alloys, these ultrafine grains are not stable at the elevated temperatures which are needed for diffusion-controlled processes such as superplasticity, thereby precluding the possibility of achieving HSR SP in these materials.
- (d) Ultrafine grain sizes may be retained at high temperatures, within the diffusion regime, when small precipitates are present within the matrix to restrict grain growth: this effect was illustrated for several alloys including a commercial Al-Mg-Li-Zr alloy and Supral-100 (Al-2004).
- (e) It is possible to achieve HSR SP in non-superplastic Al-based alloys by subjecting the materials to ECAP and then deforming in tension at temperatures above one-half of the absolute melting temperature. Remarkable results were achieved with several alloys, thereby confirming the validity of this procedure.
- (f) These results have the potential for transforming the superplastic forming industry. At present, this industry is restricted to the manufacture of low-volume high-cost components because of the low forming rates required for the fabrication process: but this may be transformed by using techniques such as ECAP to further refine the grain size into the submicrometer or nanometer range and thereby shift the strain rates associated with optimum superplasticity to much faster rates which will permit an opportunity for rapid forming.

Full details about these results, and their implications, are contained in the publications documented in the attached list. Further information is also available from the author of this report.

Participating scientific personnel:

P.B. Berbon (part-time graduate student on this program – now at Structural Metals Division, Rockwell Science Center, Thousand Oaks, CA).

S. Lee (graduate student on this program until January 2000 – scheduled to obtain Ph.D. degree during 2000).

Dr. Y. Li (Research Associate on this program – now at University of California, Irvine, CA)

Dr. B.Q. Han (Research Associate on this program until January 2000).

Publications (1997-2000)

1. T.G. Langdon, "Cavitation in High Strain Rate Superplasticity: Implications for the Flow Process," Materials Science Forum 233-234, 47-62 (1997).
2. M. Furukawa, P.B. Berbon, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon, "Production of Ultra-fine Grained Metallic Materials Using an Intense Plastic Straining Technique," Materials Science Forum 233-234, 177-184 (1997).
3. Z. Horita, M. Furukawa, M. Nemoto, N.K. Tsenev, R.Z. Valiev, P.B. Berbon and T.G. Langdon, "Processing of an Al-Mg-Li-Zr Alloy with Ultra-fine Grain Size," Materials Science Forum 243-245, 239-244 (1997).
4. Y. Li and T.G. Langdon, "A Simple Procedure for Estimating Threshold Stresses in the Creep of Metal Matrix Composites," Scripta Materialia 36, 1457-1460 (1997).
5. Y. Li and T.G. Langdon, "An Examination of Creep Data for an Al-Mg Composite," Metallurgical and Materials Transactions A 28A, 1271-1273 (1997).
6. Y. Ma and T.G. Langdon, "Creep Behavior of an Al-6061 Metal Matrix Composite Produced by Liquid Metallurgy Processing," Materials Science and Engineering A230, 183-187 (1997).
7. M. Furukawa, Z. Horita, M. Nemoto, R.Z. Valiev and T.G. Langdon, "Recrystallization of Ultrafine-Grained Materials with Non-Equilibrium Grain Boundaries," Proceedings of ReX'96: The Third International Conference on Recrystallization and Related Phenomena (T.R. McNelley, ed.), pp. 149-160. Monterey Institute of Advanced Studies, Monterey, CA (1997).
8. Y. Li and T.G. Langdon, "Creep Behavior of an Al-6061 Metal Matrix Composite Reinforced with Alumina Particulates," Acta Materialia 45, 4797-4806 (1997).
9. K. Isshiki, Z. Horita, T. Fujinami, T. Sano, M. Nemoto, Y. Ma and T.G. Langdon, "A New Miniature Mechanical Testing Procedure: Application to Intermetallics," Metallurgical and Materials Transactions A 28A, 2577-2582 (1997).
10. M. Furukawa, Y. Ma, Z. Horita, M. Nemoto, R.Z. Valiev and T.G. Langdon, "Fabrication and Properties of a Submicrometer-Grained Zn-22% Al Alloy," THERMEC'97, (T. Chandra and T. Sakai, eds.), vol. II, pp. 1875-1881. The Minerals, Metals and Materials Society, Warrendale, PA (1997).

11. M. Mabuchi, N. Saito, K. Shimojima, M. Nakanishi, Y. Yamada, M. Nakamura, T. Asahina, T.G. Langdon, H. Iwasaki and K. Higashi, "High Strain Superplasticity in Magnesium Alloys," THERMEC'97, (T. Chandra and T. Sakai, eds.), vol. II, pp. 1975-1981. The Minerals, Metals and Materials Society, Warrendale, PA (1997).
12. Y. Li and T.G. Langdon, "High Temperature Deformation of a 6061 Al Composite with Alumina Particulate Reinforcement," Proceedings of the Seventh International Conference on Creep and Fracture of Engineering Materials and Structures (J.C. Earthman and F.A. Mohamed, eds.), pp. 227-236. The Minerals, Metals and Materials Society, Warrendale, PA (1997).
13. R.Z. Valiev, D.A. Salimonenko, N.K. Tsenev, P.B. Berbon and T.G. Langdon, "Observations of High Strain Rate Superplasticity in Commercial Aluminum Alloys with Ultrafine Grain Sizes," Scripta Materialia 37, 1945-1950 (1997).
14. P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon, "Optimizing the Processing of a Commercial Al-Based Alloy for High Strain Rate Superplasticity," Microstructure, Micromechanics and Processing of Superplastic Materials: IMSP '97 (T. Aizawa, K. Higashi and M. Tokuda, eds.), pp. 45-52. Mie University Press, Tsu, Japan (1997).
15. M. Furukawa, P.B. Berbon, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon, "Age Hardening and the Potential for Superplasticity in a Fine-Grained Al-Mg-Li-Zr Alloy," Metallurgical and Materials Transactions 29A, 169-177 (1998).
16. Y. Li and T.G. Langdon, "Creep Behavior of a Reinforced Al-7005 Alloy: Implications for the Creep Processes in Metal Matrix Composites," Acta Materialia 46, 1143-1155 (1998).
17. M. Furukawa, Y. Ma, Z. Horita, M. Nemoto, R.Z. Valiev and T.G. Langdon, "Microstructural Characteristics and Superplastic Ductility in a Zn-22% Al Alloy with Submicrometer Grain Size," Materials Science and Engineering A241, 122-128 (1998).
18. P.B. Berbon, N.K. Tsenev, R.Z. Valiev, M. Furukawa, Z. Horita, M. Nemoto and T.G. Langdon, "Superplasticity in Alloys Processed by Equal-Channel Angular Pressing," Superplasticity and Superplastic Forming 1998 (A.K. Ghosh and T.R. Bieler, eds.), pp. 127-134. The Minerals, Metals and Materials Society, Warrendale, PA (1998).
19. Z. Horita, D.J. Smith, M. Nemoto, R.Z. Valiev and T.G. Langdon, "Observations of Grain Boundary Structure in Submicrometer-Grained Cu and Ni Using High-Resolution Electron Microscopy," Journal of Materials Research 13, 446-450 (1998).
20. Y. Li and T.G. Langdon, "Significance of Load Transfer in the High Strain Rate Superplasticity of Magnesium Alloy Composites," Superplasticity and Superplastic Forming 1998 (A.K. Ghosh and T.R. Bieler, eds.), pp. 189-196. The Minerals, Metals and Materials Society, Warrendale, PA (1998).

21. P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, R.Z. Valiev and T.G. Langdon, "Application of Equal-Channel Angular Pressing for Producing Superplastic Aluminum Alloys," Modeling the Mechanical Response of Structural Materials (E.M. Taleff and R.K. Mahidhara, eds.) pp. 173-180. The Minerals, Metals and Materials Society, Warrendale, PA (1998).
22. Y. Li and T.G. Langdon, "An Examination of the Effect of Processing Procedure on the Creep of Metal Matrix Composites," Materials Science and Engineering A245, 1-9 (1998).
23. H. Iwasaki, M. Mabuchi, K. Higashi and T.G. Langdon, "The Characteristics of Microcavitation in High Strain Rate Superplasticity," Materials Science and Engineering A246, 117-123 (1998).
24. P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon, "Requirements for Achieving High-Strain-Rate Superplasticity in Cast Aluminium Alloys," Philosophical Magazine Letters **78**, 313-318 (1998).
25. P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon, "Processing of Aluminum Alloys for High Strain Rate Superplasticity," Hot Deformation of Aluminum Alloys II (T.R. Bieler, L.A. Lalli and S.R. MacEwan, eds.), pp. 111-124. The Minerals, Metals and Materials Society, Warrendale, PA (1998).
26. T.G. Langdon, M. Furukawa, Z. Horita and M. Nemoto, "Using Intense Plastic Straining for High-Strain-Rate Superplasticity," JOM **50** (6) 41-45 (1998).
27. P.B. Berbon, N.K. Tsenev, R.Z. Valiev, M. Furukawa, Z. Horita, M. Nemoto and T.G. Langdon, "Fabrication of Bulk Ultrafine-Grained Materials through Intense Plastic Straining," Metallurgical and Materials Transactions 29A, 2237-2243 (1998).
28. Y. Li and T.G. Langdon, "High Strain Rate Superplasticity in Metal Matrix Composites: The Role of Load Transfer," Acta Materialia **46**, 3937-3948 (1998).
29. Y. Li and T.G. Langdon, "A Comparison of the Creep Properties of an Al-6092 Composite and the Unreinforced Matrix Alloy," Metallurgical and Materials Transactions 29A, 2523-2531 (1998).
30. Y. Li and T.G. Langdon, "Creep Behavior of Powder Metallurgy Al-6092 and Al-6061 Alloys," Hot Deformation of Aluminum Alloys II, (T.R. Bieler, L.A. Lalli and S.R. MacEwan, eds.), pp. 139-148. The Minerals, Metals and Materials Society, Warrendale, PA (1998).
31. Y. Li and T.G. Langdon, "An Examination of High Strain Rate Superplasticity in Aluminum Matrix Composites," The Third Pacific Rim International Conference on Advanced Materials and Processing (PRICM 3) (M.A. Iman, R. DeNale, S. Hanada, Z. Zhong and D.N. Lee, eds.), pp. 1785-1790. The Minerals, Metals and Materials Society, Warrendale, PA (1998).

32. P.B. Berbon, N.K. Tsenev, R.Z. Valiev, M. Furukawa, Z. Horita, M. Nemoto and T.G. Langdon, "High Strain Rate Superplasticity in Fine-Grained Commercial Al Alloys Processed by Equal-Channel Angular Pressing," Advanced Light Alloys and Composites (R. Ciach, ed.), pp. 477-486, Kluwer, Dordrecht, The Netherlands (1998).
33. Y. Li and T.G. Langdon, "Creep Properties of Metal Matrix Composites: An Examination of Recent Developments," Recent Research Developments in Materials Science **1**, 235-242 (1998).
34. Y. Li and T.G. Langdon, "Fundamental Aspects of Creep in Metal Matrix Composites," Metallurgical and Materials Transactions **30A**, 315-324 (1999).
35. Y. Li, V. Sklenicka and T.G. Langdon, "Creep Properties of an AZ91 Magnesium-Based Composite," Creep Behavior of Advanced Materials for the 21st Century (R.S. Mishra, A.K. Mukherjee and K.L. Murty, eds.), pp. 171-178. The Minerals, Metals and Materials Society, Warrendale, PA (1999).
36. Y. Li and T.G. Langdon, "An Examination of a Substructure-Invariant Model for the Creep of Metal Matrix Composites," Materials Science and Engineering A265, 276-284 (1999).
37. R.Z. Valiev, R.K. Islamgaliev, N.F. Kuzmina, Y. Li and T.G. Langdon, "Strengthening and Grain Refinement in an Al-6061 Metal Matrix Composite through Intense Plastic Straining," Scripta Materialia **40**, 117-122 (1999).
38. T.G. Langdon, "An Examination of Flow Processes in High Strain Rate Superplasticity," Materials Science Forum **304-306**, 13-20 (1999).
39. Y. Li and T.G. Langdon, "Creep Behavior of an AZ91 Magnesium Alloy Reinforced with Alumina Fibers," Metallurgical and Materials Transactions **30A**, 2059-2066 (1999).
40. Y. Li and T.G. Langdon, "Characteristics of Creep Deformation in Discontinuously Reinforced Metal Matrix Composites," Materials Science and Technology **15**, 357-365 (1999).
41. Y. Li and T.G. Langdon, "Creep Properties of Metal Matrix Composites," Creep Behavior of Advanced Materials for the 21st Century (R.S. Mishra, A.K. Mukherjee and K.L. Murty, eds.), pp. 73-82. The Minerals, Metals and Materials Society, Warrendale, PA (1999).
42. S. Lee, P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon, "Developing Superplastic Properties in an Aluminum Alloy through Severe Plastic Deformation," Materials Science and Engineering A227, 63-72 (1999).
43. Y. Li and T.G. Langdon, "A Unified Interpretation of Threshold Stresses in the Creep and High Strain Rate Superplasticity of Metal Matrix Composites," Acta Materialia **47**, 3395-3403 (1999).

44. T.G. Langdon, "Recent Developments in High Strain Rate Superplasticity," Materials Transactions JIM **40**, 716-722 (1999).
45. B.Q. Han and T.G. Langdon, "Creep Behavior of an Al-7005 Alloy Reinforced with Alumina Particulates," Advanced Materials for the 21st Century: The 1999 Julia R. Weertman Symposium (Y.-W. Chung, D.C. Dunand, P.K. Liaw and G.B. Olsen, eds.), pp. 129-136. The Minerals, Metals and Materials Society, Warrendale, PA (1999).
46. Y. Li and T.G. Langdon, "Equal-Channel Angular Pressing of an Al-6061 Metal Matrix Composite," Journal of Materials Science (in press).
47. Z. Horita, M. Furukawa, M. Nemoto, R.Z. Valiev and T.G. Langdon, "Characterization of Ultrafine-Grained Structures Produced by Severe Plastic Deformation," Investigations and Applications of Severe Plastic Deformation, Kluwer, Dordrecht, The Netherlands (in press).

REPORT DOCUMENTATION PAGE			Form Approved OMB NO. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comment regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE January 31, 2000		3. REPORT TYPE AND DATES COVERED Final Report (August 1996 - January 2000)
4. TITLE AND SUBTITLE A Critical Evaluation of the Factors Influencing High Strain Rate Superplasticity			5. FUNDING NUMBERS DAAH04-96-1-0332	
6. AUTHOR(S) Terence G. Langdon				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) University of Southern California Los Angeles, CA 90089-1453			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211			10. SPONSORING / MONITORING AGENCY REPORT NUMBER ARO 34391.49-ms	
11. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12 b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) This Final Report lists the publications arising from this research program and also provides a brief description of the major findings. There were two major objectives of this program and both were fulfilled within the research period. These objectives were (1) to obtain a detailed understanding of the creep properties of metal matrix composites which are important in the field of high strain rate superplasticity and (2) to attempt to extend the range of materials exhibiting high strain rate superplasticity to commercial unreinforced alloys by using a new processing technique in which an ultrafine grain size is achieved by introducing severe plastic deformation. This latter objective has led to the first demonstration of superplasticity at very high strain rates and at relatively low temperatures in conventional materials.				
14. SUBJECT TERMS superplasticity, processing, creep			15. NUMBER OF PAGES 9	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL	